



(19)

Europäisches Patentamt

European Patent Office

Office européen des brevets



(11)

EP 0 846 666 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:

18.08.2004 Bulletin 2004/34

(51) Int Cl.⁷: **C04B 28/18**

(86) International application number:
PCT/JP1996/001757

(21) Application number: **96918905.9**

(87) International publication number:
WO 1997/049645 (31.12.1997 Gazette 1997/57)

(22) Date of filing: **25.06.1996**

(54) PROCESS OF PRODUCING A CALCIUM SILICATE PLATE

VERFAHREN ZUR HERSTELLUNG EINER KALZIUMSILIKATPLATTE

METHODE DE FABRICATION D'UNE PLAQUE DE SILICATE DE CALCIUM

(84) Designated Contracting States:
BE FR GB

(43) Date of publication of application:
10.06.1998 Bulletin 1998/24

(60) Divisional application:
03006437.2 / 1 338 579

(73) Proprietor: **A & A Material Corporation**
Tsurumi-ku, Yokohama-shi (JP)

(72) Inventors:

- **3UZUKI, Seishiro, ASK Corporation**
Yokohama-shi, Kanagawa 230 (JP)
- **SAKIYAMA, Masato,**
ASK Corp. Central Res. Lab.
Ishiooka-shi, Ibaraki 315 (JP)
- **ASAMI, Takuya, ASK Corp. Central Res. Lab.**
Ishiooka-shi, Ibaraki 315 (JP)
- **IWANAGA, Tomoki, ASK Corp. Central Res. Lab.**
Ishiooka-shi, Ibaraki 315 (JP)
- **OSHIO, Yasuhide, ASK Corp. Central Res. Lab.**
Ishiooka-shi, Ibaraki 315 (JP)

- **SHIROMOTO, Shigemitsu,**
ASK Corp. Central Res. Lab.
Ishiooka-shi, Ibaraki 315 (JP)
- **SUGIYAMA, Toru, ASK Corp. Central Res. Lab.**
Ishiooka-shi, Ibaraki 315 (JP)
- **ODA, Masaaki, ASK Corp. Central Res. Lab.**
Ishiooka-shi, Ibaraki 315 (JP)

(74) Representative: **Waldren, Robin Michael et al**
MARKS & CLERK,
57-60 Lincoln's Inn Fields
London WC2A 3LS (GB)

(56) References cited:

- | | |
|-------------------------|-------------------------|
| EP-A- 0 678 488 | JP-A- 49 008 518 |
| JP-A- 52 105 926 | JP-A- 52 135 330 |
| US-A- 4 128 434 | US-A- 4 144 121 |
- **PATENT ABSTRACTS OF JAPAN vol. 018, no. 625 (C-1279), 29 November 1994 (1994-11-29) & JP 06 239672 A (NKK CORP), 30 August 1994 (1994-08-30)**
 - **PATENT ABSTRACTS OF JAPAN vol. 017, no. 436 (C-1096), 12 August 1993 (1993-08-12) & JP 05 097498 A (NICHIA CORP), 20 April 1993 (1993-04-20)**

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

EP 0 846 666 B1

Description

INDUSTRIAL FIELD OF THE INVENTION

5 [0001] The present invention relates to a method of manufacturing a calcium silicate board.

RELATED ART AND PROBLEMS

10 [0002] Conventionally, calcium silicate boards have been widely used for building materials, in particular, for interior materials because of such characteristics as being light, excellent in workability and dimensional stability, and non-combustible.

[0003] Calcium silicate boards used for building material are formed using a forming process such as the Hatschek sheet machine process a press mold process or a mono-layer mold process and are manufactured as follows: a molded body is formed from a calcareous material, a silica containing material, reinforcing fibers and fillers, such as light-weight aggregate, then, in general, subjected to curing reaction by saturated water vapor in a pressure container. Generally, the bulk specific gravity of the calcium silicate boards is in a range of from 0.7 to 1.2.

[0004] Since calcium silicate boards are used as an interior material, such as ceiling materials and wall materials, lightening the boards is desirable, however, according to the above methods it is necessary to mix a considerable amount of light-weight aggregate so as to achieve a bulk specific gravity of 0.5 to 0.8, resulting in a decrease in the specific strength of the boards.

[0005] In particular, when a light-weight calcium silicate board having a bulk specific gravity of 1.0 or less is manufactured according to the Hatschek sheet machine process, the following problems sometimes occur: because of the low strength and large water content of the molded body before hydrothermal reaction, the redundant water thermally expands during hydrothermal reaction or the vapor pressure increases, resulting in interlayer peeling or bursting.

25 [0006] To avoid the above problems, in some methods, a molded body is pressed after forming then subjected to hydrothermal reaction or a molded body is tightened by a turn-buckle during the hydrothermal reaction. However, in accordance with these methods, such problems occur that the bulk specific gravity increases and more labor is required for manufacturing.

[0007] In addition, Japanese Patent Laid-Open No. 6-287083 discloses a method of removing the redundant water, though this method cannot be commonly used because a specific pressure container or a vapor heater is necessary.

[0008] Further, conventionally, a material slurry containing a calcareous material and an amorphous silica containing material is heated at a normal pressure for gelation so as to achieve a light-weight board. However, according to this method, the board strength is inevitably reduced, and further, the material is so soft that it is readily shaved during polishing. Therefore, cellulose fibers become extremely fuzzy such that the surface smoothness is impaired when the board is used after coating.

[0009] Moreover, another method is disclosed in Japanese Patent Laid-Open Nos. 52-105926 and 52-135330, wherein calcium silicate boards exhibiting excellent strength are obtained by adding a xontlite slurry, which is a kind of calcium silicate hydrate, or a crystalline calcium slurry. Since productivity decreases according to these methods, improvement is required from a viewpoint of manufacturing costs.

40 [0010] Additionally, in Japanese Patent Laid-Open Nos. 5-213657 and 7-41354, the inventors of the present invention have already disclosed a method of adding both a gelatinized slurry, comprising a calcareous material and an amorphous silica containing material, and a crystalline calcium silicate hydrate slurry. However, in the case of a board laminated by the Hatschek sheet machine process, some problems, such as interlayer peeling or bursting, occur even in this method of adding gels.

45 [0011] Furthermore, Japanese Patent Publication No. 58-30259 discloses a method of manufacturing a calcium silicate molded body characterized by the following: a silica containing material and a calcareous material are dispersed in water of which the amount by weight is 15 times or more than that of the materials as a solid component; the resultant mixture is heat-reacted at 130°C or more under a pressurized condition so as to form an aqueous slurry, which has a sedimentation volume of 15 cm³/g or more, contains calcium silicate composed of C-S-H or tobermorite; to the aqueous slurry asbestos is added and subjected to dewatering and forming; and the calcium silicate hydrate is transformed by water vapor curing under a pressurized condition. However, the calcium silicate molded body obtained by this method is used as a heat storing material or a heat insulating material, and according to an embodiment of this Publication, the bulk specific gravity thereof is as small as 0.1 to 0.2 g/cm³. Thus the calcium silicate molded body cannot be employed for a calcium silicate board used as a building material.

55 [0012] An object of the present invention is to provide a method of manufacturing a calcium silicate board having a bulk specific gravity of 1.0 or less, in which the foregoing problems are solved such that peeling or bursting during the hydrothermal reaction is eliminated because the strength of a molded body (green sheet) is improved.

[0013] The invention does this by using a material having an excellent reactivity with a calcareous material and by

primary curing before the hydrothermal reaction.

[0014] The present invention relates to a method of manufacturing a light-weight calcium silicate board (hereinafter referred to as a calcium silicate board) comprising a step of forming a material slurry into a prescribed shape, the material slurry comprising, as a solid component, 17 to 50wt% of calcareous material, 15 to 45wt% of silica containing material, 2 to 8 wt% of fiber material and 5 to 40wt% of inorganic fillers, and a step of hydrothermally reacting the obtained molded body in a pressure container, wherein 2 to 20wt% of at least one kind of amorphous silica containing material and/or silicate material, each having a specific surface area of 1 m²/g or more, is used as a portion of the silica containing material; and, before the hydrothermal reaction, the molded body is subjected to primary curing under conditions where (curing temperature - 15) x curing time ≥ 120°C·hr.

[0015] A method of manufacturing a calcium silicate board according to the present invention will now be described, by way of example.

[0016] Calcium hydroxide and quicklime are examples of a calcareous material. Silica sand and sintered diatomaceous earth are examples of a crystalline silica containing material. When the Ca/(Si + Al) molar ratio of the foregoing slurry is less than 0.3, effects on decreasing the specific gravity and increasing the matrix strength are undesirably reduced because of a small production amount of calcium silicate hydrate. Whereas if the ratio exceeds 0.8, undesirably, the moisture content is raised because of an increase in sedimentation volume, thus impairing productivity.

[0017] In addition, silica sand, flay ash, diatomaceous earth, silicon dust, white carbon, zeolite, and montmorillonite are examples of a silica containing material.

[0018] Moreover, cellulose fibers, polypropylene, vinylon, glass fibers and carbon fibers are examples of a fiber material.

[0019] Further, perlite, wallastonite, mica, talc, calcium carbonate and gypsum are examples of inorganic fillers.

[0020] In addition, as a forming method, any conventional forming process, including the Hatschek sheet machine process, mold press process, and mono-layer mold process, can be used.

[0021] The fundamental position of a material slurry used for a method of manufacturing a calcium silicate board according to the present invention is a conventional composition, as follows: as a solid component, 17 to 50 wt% of calcareous material, 15 to 45 wt% of silica containing material; 2 to 8 wt% of fiber material; and 5 to 40% of inorganic fillers.

[0022] The same materials as above can be used as a calcareous material. A calcareous material content of less than 17 wt% or more than 50 wt% is not preferable because it causes a decrease in the flexural strength and an increase in the dimensional change rate due to water absorption.

[0023] In addition, silica sand, flay ash, etc., can be used as a silica containing material. A silica containing material content of less than 15wt% or more than 45wt% is not preferable because it causes a decrease in the flexural strength and an increase in the dimensional change rate due to water absorption.

[0024] Moreover, the same materials as above can be used as a fiber material. A fiber material content of less than 2wt% is not preferable because it decreases the flexural strength and a fiber material content of more than 8wt% undesirably causes a loss of the non-combustibility. Additionally, when polypropylene, vinylon, glass fibers or carbon fibers are used, it is necessary to set the content thereof to 5wt% or less.

[0025] Further, the same materials as mentioned above can be used as inorganic fillers. An inorganic filler content of less than 5wt% is not preferable because it causes an increase in the dimensional change rate due to water absorption. An inorganic filler content of more than 40wt% is not preferable since it decreases the flexural strength.

[0026] The first novel characteristic of the present invention is that an amorphous silica containing material or a silicate material of which the specific surface area is 1 m²/g or more is used as a portion of a silica containing material of a material slurry having the foregoing composition. "The specific surface area" in this description is estimated by the gaseous N₂ absorption method. It is not preferable that the specific surface area of the amorphous silica containing material or the silicate material be less than 1 m²/g, since a green sheet cannot obtain sufficient strength before the hydrothermal reaction due to poor reactivity.

[0027] Further, the content of the amorphous silica containing material or the silicate material having a specific surface area of 1 m²/g or more is in a range of from 2 to 20wt%. A content of less than 2wt% is not preferable because of poor strength reproducibility, and a content of more than 20wt% is not preferable because it reduces the freeness, resulting in a decrease in productivity. When a material having a specific surface area of 1 to 10 m²/g is used, the preferable content thereof is 10wt% or more, in the case of a material having a specific surface area of 10 to 100 m²/g, the preferable content thereof is 5wt% or more, and in the case of a material having a specific surface area of 100 m²/g or more, the preferable content thereof is 2wt% or more.

[0028] Examples of an amorphous silica containing material having a specific surface area of 1 m²/g or more are diatomaceous earth, silicon dust, fly ash, and white carbon. Additionally, examples of a silicate material having a specific surface area of 1 m²/g or more are zeolite, pyrophyllite, allophane, montmorillonite mineral, chlorite mineral, and attapulgite. Two or more kinds of these materials can be used together.

[0029] The second novel characteristic of the present invention is that a green sheet (molded body) is not subjected

to the hydrothermal reaction as it is but undergoes primary curing, which green sheet is obtained such that a material slurry composed of the foregoing material compositions is formed into a prescribed shape by, for instance, the Hatschek sheet machine process. Primary curing proceeds under the following conditions: (curing temperature - 15) x curing time $\geq 120^{\circ}\text{C}\cdot\text{hr}$. The primary curing conditions of less than $120^{\circ}\text{C}\cdot\text{hr}$ are not preferable because a green sheet cannot obtain sufficient strength due to a shortage of curing. Primary curing requires a curing temperature of more than 15°C and, preferably, it proceeds under conditions of $240^{\circ}\text{C}\cdot\text{hr}$ or more in a curing temperature range of 30 to 80°C .

[0030] After the afore-mentioned primary curing, the green sheet may be subjected to a conventional hydrothermal reaction and formed into a calcium silicate board through conventional processes. The hydrothermal reaction can proceed in a pressure container under saturated water vapor at a temperature of 150 to 200°C and, preferably at 170 to 190°C for 5 to 20 hours and, more preferably, for 8 to 12 hours.

[0031] According to a manufacturing method incorporated in the present invention, a calcium silicate board having a bulk specific gravity of 1.0 or less can be obtained.

EXAMPLES

[0032] A method of manufacturing a calcium silicate board according to the present invention will be better understood in detail from the following description taken in conjunction with the examples and comparative examples.

Example 1 and Comparative Example 1

[0033] Manufacturing examples of calcium silicate hydrate slurries essentially consisting of an amorphous material:

[0034] Slurries essentially consisting of an amorphous material were obtained as follows: calcium hydroxide and silica sand were blended according to formulations shown in Table 1; each of the mixtures was mixed with 13 times the amount of water and stirred; then each of the resultant mixtures was subjected to a hydrothermal reaction according to the corresponding conditions shown in Table 1. An autoclave for experimental use was employed for the hydrothermal reaction.

TABLE 1

	Experimental No.					
	1	2	3	4	5	6
Materials	Silica Sand Calcium Hydroxide					
Ca/(Si+Al) Molar Ratio	0.4	0.6	0.8	0.2	1.0	0.6
Temp. ($^{\circ}\text{C}$)	180	180	150	180	150	180
Time (hr.)	6	4	4	6	4	10
Water Ratio	13	13	13	13	13	13
Stirring Rate (m/min)	100	100	100	100	100	100
Reaction Rate of Crystalline Silica Containing Material	48	57	78	31	95	73
Sedimentation Volume (ml/g)	6.4	7.1	9.0	5.8	14.5	15.8

[0035] Materials were prepared according to formulations shown in Table 2. Each of the materials was formed into a slurry having a solid content of 10%, press-molded into 30 by 30 by 0.8 cm pieces at a press pressure of 10 kg/cm^2 , then subjected to a hydrothermal reaction for 10 hours at 180°C under a saturated vapor pressure.

[0036] Table 2 shows the bulk specific gravity, the flexural strength (under absolute dry conditions) and abrasion index.

Table 2

		Examples				Comparative Examples				
		1	2	3	4	1	2	3	4	5
Formulation (wt%)	Calcium Carbonate Hydrate Slurry									
	Experimental No.	1	2	3	1	2	4	5	6	2
	Content (As a Solid Component)	28	7	15	7	35	28	15	7	3
	Calcium Hydroxide	32	28	29	34	26	35	29	28	41
	Silica Sand	24	26	28	33	23	21	28	26	40
	Wallastonite	10	20	10	10	10	10	10	20	10
	Perlite	-	5	-	-	-	-	-	5	-
	Calcium Carbonate	-	10	10	10	-	-	10	10	-
	Pulp	5	3	7	5	5	5	7	3	5
	Glass Fiber	1	1	1	1	1	1	1	1	1
Bulk Specific Gravity		0.55	0.68	0.63	0.78	0.48	0.73	0.61	0.64	0.82
Flexural Strength (kg/cm ²)		92	115	122	118	88	104	111	118	126
Abrasion Index		1.8	0.43	1.1	0.32	2.3	0.27	1.4	1.2	0.20
Productivity		o	o	Δ	o	x	o	x	x	o

[0037] In Table 2, the filtration rate (ml/sec) is defined as the value of filtered water (ml)/filtration time (second) when one liter of slurry, of which the concentration is 4.2% as a solid component, is put in a Büchner funnel having a diameter of 16 cm and filtered at 50 cmHg. The filtration rate of 30 or more is indicated by o, that of 20 or more and less than 30 is indicated by Δ, and that of less than 20 is indicated by ×.

Example 2 and Comparative Example 2

[0038] Calcium hydroxide, silica sand, wallastonite, perlite, pulp, an amorphous silica containing material and a silicate material were blended according to formulations shown in Table 3, then each of the mixtures was mixed with 12 times the amount of water and stirred.

[0039] By further adding water thereto, material slurries of which the solid content was approximately 3wt% were obtained and formed into 6 mm thick sheets by the Hatschek sheet machine process.

[0040] Then, each of the resultant green sheets was subjected to primary curing according to the conditions, i. e., the temperature and time, shown in Table 3, followed by the hydrothermal reaction for 10 hours at 180°C under a saturated vapor pressure.

[0041] Table 3 shows peeling and bursting of the green sheets after the hydrothermal reaction, and also, the bulk specific gravity and the flexural strength of the obtained calcium silicate boards under absolute dry conditions.

Table 3

	Examples										Comparative Examples				
	1	2	3	4	5	6	7	8			1	2	3	4	5
Formulation (%)	Calcium Hydroxide	40	40	40	29	38	38	36			38	40	45	40	24
	Silica Sand	26	30	33	27	23	29	23			35	26	32	16	15
	Perlite	4	6	6	6	6	6	6			7	4	7	4	6
	Wollastonite	10	10	10	10	10	10	15			15	10	10	10	20
	Pulp	5	5	5	5	5	5	5			5	5	5	5	5
	Diatomaceous Earth	15	-	-	-	-	-	-			-	15	-	25	-
	Silicon Dust	-	9	-	-	-	-	-			-	-	-	-	-
	White Carbon	-	-	6	-	-	-	-			-	-	1	-	-
	Zeolite	-	-	-	12	-	-	-			-	-	-	-	-
	Calcium Carbonate	-	-	-	-	15	-	-			-	-	-	-	20
Primary Curing	Montmorillonite	-	-	-	-	10	-	-			-	-	-	-	10
	Allophane	-	-	-	-	-	18	-			-	-	-	-	-
	Attapulgite	-	-	-	-	-	-	-			-	-	-	-	-
	Chlorite	-	-	-	-	-	-	15			-	-	-	-	-
Temp. (°C)	60	40	25	70	60	40	70	60			60	20	25	60	60
	8	24	12	10	8	24	10	8			8	10	12	8	8
	360	600	120	550	360	600	550	360			360	50	120	360	360
Flexural Strength (kg/cm ²)	12	13	10	21	11	14	17	18			4	4	5	17	10
	-	-	-	-	-	-	-	-			+	+	+	-	-
Peeling	-	-	-	-	-	-	-	-			+	+	+	-	-
Productivity	0	0	0	0	0	0	0	0			0	0	0	x	0
Bulk Specific Gravity	0.73	0.80	0.81	0.84	0.77	0.75	0.84	0.83			0.76	0.73	0.83	0.70	0.95
Flexural Strength (kg/cm ²)	111	127	133	132	108	105	128	126			121	117	135	104	91

[0042] In Table 3, the specific surface area estimated by the gaseous N₂ absorption method is 2.0 m²/g for diatomaceous earth, 20 m²/g for silicon dust, 220 m²/g for white carbon, and 6.5 m²/g for zeolite. In addition, O indicates excellent productivity and × indicates inferior productivity.

5 Example 3 and Comparative Example 3

[0043] Each of the materials prepared according to formulations shown in Table 4 was mixed with 12 times the amount of water and stirred. The gel used here was obtained by gelation of calcium hydroxide and diatomaceous earth at a weight ratio of 1:1, which gelation proceeded at a water ratio of 5 for 2 hours at 90°C. In the Table, the gel content is shown as a solid component.

[0044] By further adding water thereto, material slurries of which the solid content was approximately 3wt% were obtained and formed into 6 mm thick sheets by the Hatschek sheet machine process.

[0045] Then, each of the resultant green sheets was subjected to primary curing according to the conditions, i. e., the temperature and time, shown in Table 4, followed by a hydrothermal reaction in a pressure container for 10 hours at 180°C under a saturated vapor pressure.

[0046] Table 4 shows the flexural strength of molded bodies immediately after primary curing, the bulk specific gravity, the flexural strength, and interlayer peel strength after the hydrothermal reaction, wherein the latter two are evaluated under absolute dry conditions.

20

25

30

35

40

45

50

55

TABLE 4

	Examples										Comparative Examples				
	1	2	3	4	5	6	7	8			1	2	3	4	5
Formulation (%)															
Calcium Hydroxide	32	30	17	17	23	31	33	31			32	28	15	30	19
Silica Sand	26	24	27	27	16	15	24	18			26	24	24	24	10
Silicon Dust	6	-	-	-	-	-	-	-			6	-	-	-	-
Zeolite	-	15	-	-	-	-	-	-			-	15	-	15	-
Montmorillonite	-	-	-	-	10	-	-	-			-	-	-	-	10
Allophane	-	-	-	-	-	18	-	-			-	-	-	-	-
Attapulgite	-	-	-	-	-	-	12	-			-	-	-	-	-
Chlorite	-	-	-	-	-	-	-	15			-	-	-	-	-
Perlite	-	5	-	-	5	-	5	-			-	5	-	5	5
Wallastonite	10	10	10	10	15	10	10	10			10	10	10	10	20
Pulp	5	5	5	5	5	5	5	5			5	7	5	5	5
Glass Fiber	1	1	1	1	1	1	1	1			1	1	1	1	1
Portland Cement	-	-	20	20	-	-	-	-			-	-	25	-	-
Calcium Carbonate	-	-	-	-	15	-	-	-			-	-	-	-	20
Gel	20	10	20	20	10	20	10	20			20	10	20	10	10
Primary Curing															
Temp. (°C)	50	70	50	15	60	40	70	60			-	30	30	15	60
Time (Hr.)	12	3	8	24	8	24	10	8				8	8	24	8
Flexural Strength Immediately After	4.8	4.5	4.8	4.9	4.6	4.5	4.8	4.4			4.8	6.4	5.1	4.4	4.6
Flexural Strength After Primary Curing: (2)	7.1	12.0	8.5	7.2	10.8	7.4	12.4	13.8			5.0	7.1	7.0	5.5	9.9
(2)/(1)	1.48	2.67	1.77	1.47	2.35	1.64	2.58	3.14			1.04	1.11	1.37	1.25	2.15
Bulk Specific Gravity	0.63	0.65	0.69	0.69	0.66	0.63	0.65	0.64			0.63	0.65	0.72	0.65	0.66
Flexural Strength: (3) (kg/cm ²)	84	83	98	92	90	82	87	86			68	92	103	80	88
Interlayer Peel Strength: (4) (kg/cm ²)	3.1	4.9	7.9	5.0	4.8	3.4	7.2	6.8			0.3	0.9	6.7	0.8	4.6
(4)/(3) (%)	3.7	5.9	8.1	5.4	5.3	4.1	8.3	7.9			0.44	0.98	6.5	1.00	5.2
Peeling During Hydrothermal Reaction	-	-	-	-	-	-	-	-			+	+	-	+	-

[0047] In Table 4, the specific surface area estimated by the gaseous N₂ absorption method is 20 m²/g for silicon dust, 6.5 m²/g for zeolite, and 2.0 m²/g for diatomaceous earth.

ADVANTAGE

[0048] By a method of manufacturing a calcium silicate board according to the present invention, it is possible to

improve the strength of the green sheet obtained by forming a material slurry into a prescribed shape, thereby preventing the green sheet from peeling, bursting or the like during the hydrothermal reaction.

5 Claims

1. A method of manufacturing a light-weight calcium silicate board comprising;
a step of forming a material slurry into a prescribed shape, said material slurry comprising, as a solid component, 17 to 50 wt% of calcareous material, 15 to 45 wt% of silica containing material, 2 to 8 wt% of fiber material and 5 to 40 wt% of inorganic fillers; and
a step of hydrothermally reacting the obtained molded body in a pressure container, wherein 2 to 20 wt% of at least one kind of amorphous silica containing material and/or silicate material, each having a specific surface area of 1 m²/g or more, is used as a portion of said silica containing material; and, before said hydrothermal reaction, said molded body is subjected to primary curing under conditions where (curing temperature -15) x curing time $\geq 120^{\circ}\text{C}\cdot\text{hr}$.
2. A method according to claim 1, in which the fiber material comprises polypropylene, vinylon, glass fibers or carbon fibers, and its content in the slurry is 5 wt% or less.
3. A method according to claim 1 or 2, in which the silica containing material has a specific surface area of 1 to 10 m²/g and its content in the slurry is 10 wt% or more.
4. A method according to claim 1 or 2, in which the silica containing material has a specific surface area of 10 to 104 m²/g and its content in the slurry is 5 wt% or more.
5. A method according to claim 1 or 2, in which the silica containing material has a specific surface area of 100 m²/g or more and its content in the slurry is 2 wt% or more.
6. A method according to any preceding claim, in which the primary curing is at a curing temperature of 30° to 80°C and (curing temperature -15) x curing time $\geq 240^{\circ}\text{C}\cdot\text{hr}$.
7. A method according to any preceding claim, in which the hydrothermal reaction in the pressure container proceeds under saturated water vapour at a temperature of 150° to 200°C for 5 to 20 hours.
8. A method according to claim 7, in which the hydrothermal reaction proceeds at 170° to 190°C.

Patentansprüche

1. Verfahren zur Herstellung einer Calciumsilicat-Leichtbauplatte, wobei das Verfahren aufweist:
einen Schritt zum Formen einer Materialaufschlämmung in eine vorgeschriebene Gestalt, wobei die Materialaufschlämmung als Feststoffkomponente 17 bis 50 Gew.-% kalkhaltiges Material, 15 bis 45 Gew.-% silicahaltiges Material, 2 bis 8 Gew.-% Fasermaterial und 5 bis 40 Gew.-% anorganische Füllstoffe aufweist; und
einen Schritt zur hydrothermalen Reaktion des erhaltenen Formkörpers in einem Druckbehälter, wobei 2 bis 20 Gew.-% mindestens einer Art eines amorphen silicahaltigen Materials und/oder Silicatmaterials, das jeweils eine spezifische Oberfläche von 1 m²/g oder mehr aufweist, als Teil des silicahaltigen Materials eingesetzt wird und der Formkörper vor der hydrothermalen Reaktion einer Primäraushärtung unter Bedingungen mit (Aushärtungstemperatur - 15) × Aushärtungszeit $\geq 120^{\circ}\text{C}\cdot\text{h}$ ausgesetzt wird.
2. Verfahren nach Anspruch 1, wobei das Fasermaterial Polypropylen, Vinylon, Glasfasern oder Kohlefasern aufweist und sein Anteil in der Aufschlämmung höchstens 5 Gew.-% beträgt.
3. Verfahren nach Anspruch 1 oder 2, wobei das silicahaltige Material eine spezifische Oberfläche von 1 bis 10 m²/g aufweist und sein Anteil in der Aufschlämmung mindestens 10 Gew.-% beträgt.
4. Verfahren nach Anspruch 1 oder 2, wobei das silicahaltige Material eine spezifische Oberfläche von 10 bis 100

m²/g aufweist und sein Anteil in der Aufschlammung mindestens 5 Gew.-% beträgt.

- 5 5. Verfahren nach Anspruch 1 oder 2, wobei das silicahaltige Material eine spezifische Oberfläche von mindestens 100 m²/g aufweist und sein Anteil in der Aufschlammung mindestens 2 Gew.-% beträgt.
6. Verfahren nach einem der vorstehenden Ansprüche, wobei die Primäraushärtung bei einer Aushärtungstemperatur von 30° bis 80°C erfolgt und (Aushärtungstemperatur - 15) x Aushärtungszeit ≥ 240°C·h gilt.
- 10 7. Verfahren nach einem der vorstehenden Ansprüche, wobei die hydrothermale Reaktion im Druckbehälter unter gesättigtem Wasserdampf bei einer Temperatur von 150°C bis 200°C über 5 bis 20 Stunden abläuft.
8. Verfahren nach Anspruch 7, wobei die hydrothermale Reaktion bei 170° bis 190°C abläuft.

15 Revendications

1. Procédé de fabrication d'un panneau de faible poids en silicate de calcium, comprenant :

20 une étape de façonnage d'une suspension de matériaux en une forme prédéterminée, ladite suspension de matériaux comprenant, en tant que constituant solide, 17 à 50 % en poids d'un matériau calcaire, 15 à 45 % en poids d'un matériau contenant de la silice, 2 à 8 % en poids d'un matériau fibreux et 5 à 40 % en poids de charges minérales ; et
une étape de réaction hydrothermique du corps moulé obtenu dans un récipient sous pression,

25 dans lequel 2 à 20 % en poids d'au moins une sorte d'un matériau contenant de la silice amorphe et/ou d'un matériau de silicate, chacun ayant une surface spécifique de 1 m²/g ou plus, sont utilisés en tant que partie dudit matériau contenant de la silice ; et, avant ladite réaction hydrothermique, ledit corps moulé est soumis à un durcissement primaire dans des conditions où (température de durcissement - 15) x temps de durcissement ≥ 120°C.h.

- 30 2. Procédé selon la revendication 1, dans lequel le matériau fibreux comprend du polypropylène, du vinylon, des fibres de verre ou des fibres de carbone, et sa teneur dans la suspension est de 5 % en poids ou moins.
3. Procédé selon la revendication 1 ou 2, dans lequel le matériau contenant de la silice a une surface spécifique de 1 à 10 m²/g et sa teneur dans la suspension est de 10 % en poids ou plus.
- 35 4. Procédé selon la revendication 1 ou 2, dans lequel le matériau contenant de la silice a une surface spécifique de 10 à 100 m²/g et sa teneur dans la suspension est de 5 % en poids ou plus.
5. Procédé selon la revendication 1 ou 2, dans lequel le matériau contenant de la silice a une surface spécifique de 100 m²/g ou plus et sa teneur dans la suspension est de 2 % en poids ou plus.
- 40 6. Procédé selon l'une quelconque des revendications précédentes, dans lequel le durcissement primaire est à une température de durcissement de 30° à 80°C et (température de durcissement - 15) x temps de durcissement ≥ 240°C.h.
- 45 7. Procédé selon l'une quelconque des revendications précédentes, dans lequel la réaction hydrothermique dans le récipient sous pression se déroule sous vapeur d'eau saturée à une température de 150° à 200°C pendant 5 à 20 heures.
- 50 8. Procédé selon la revendication 7, dans lequel la réaction hydrothermique se déroule à 170° à 190°C.